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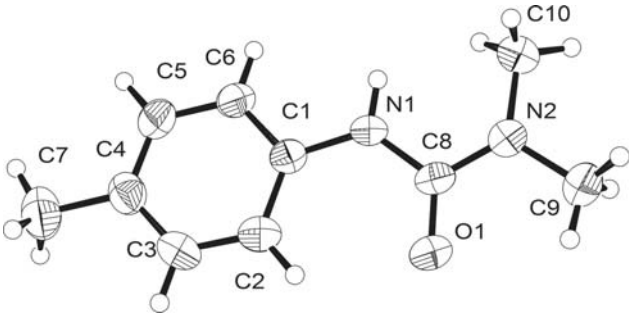
Crystal structure of 1,1-dimethyl-3-(4-methylphenyl)urea, $C_{10}H_{14}N_2O$



DOI 10.1515/ncrs-2016-0290
Received September 30, 2016; accepted January 13, 2017; available online February 21, 2017

Abstract

$C_{10}H_{14}N_2O$, monoclinic, *Cc* (no. 9), $a = 6.8354(9)$ Å, $b = 13.8221(17)$ Å, $c = 10.2905(12)$ Å, $\beta = 90.625(11)°$, $V = 972.2(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0526$, $wR_{ref}(F^2) = 0.1335$, $T = 296(2)$ K.

CCDC no.: 1527333

The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesised by reaction of equimolar quantities of 4-methylaniline and dimethylcarbamoyl chloride in dichloromethane at 40 °C for 1 h in the presence of excess triethylamine (1.38 mole equivalents). The crude product was recrystallized from a 1:3 mixture (by volume) of ethyl acetate and diethyl ether to give 1,1-dimethyl-3-(4-methylphenyl)urea (92%) as colourless crystals, Mp.: 141–142 °C (lit. 137–138 °C [1]; 152–153 °C [2]).

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Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.45 × 0.35 × 0.10 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.8 cm^{-1}
Diffractometer, scan mode:	SuperNova, ω-scans
$2\theta_{max}$, completeness:	59.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3791, 3791, 0.059
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2943
$N(param)_{refined}$:	120
Programs:	CrysAlis^{PRO} [17], SHELX [18], PLATON [19]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0013(4)	0.6008(2)	0.5771(3)	0.0391(7)
C2	−0.1240(5)	0.6047(3)	0.4684(4)	0.0517(10)
H2	−0.0968	0.5687	0.3945	0.062*
C3	−0.2885(5)	0.6629(3)	0.4726(4)	0.0584(11)
H3	−0.3714	0.6651	0.4004	0.070*
C4	−0.3341(4)	0.7180(3)	0.5803(4)	0.0489(9)
C5	−0.2091(4)	0.7138(3)	0.6860(4)	0.0489(8)
H5	−0.2366	0.7502	0.7596	0.059*
C6	−0.0424(4)	0.6559(3)	0.6848(4)	0.0452(8)
H6	0.0402	0.6543	0.7571	0.054*
C7	−0.5184(5)	0.7786(3)	0.5819(6)	0.0691(13)
H7A	−0.6305	0.7372	0.5721	0.104*
H7B	−0.5158	0.8242	0.5117	0.104*
H7C	−0.5259	0.8126	0.6630	0.104*
C8	0.2587(4)	0.4929(2)	0.4877(4)	0.0392(7)
C9	0.5152(6)	0.3823(3)	0.4257(5)	0.0587(10)
H9A	0.4308	0.3757	0.3511	0.088*
H9B	0.5441	0.3195	0.4608	0.088*
H9C	0.6347	0.4134	0.4007	0.088*
C10	0.5180(5)	0.4470(4)	0.6474(5)	0.0591(11)
H10A	0.6537	0.4311	0.6369	0.089*
H10B	0.4598	0.4025	0.7072	0.089*
H10C	0.5068	0.5116	0.6805	0.089*
N1	0.1702(4)	0.5411(2)	0.5869(3)	0.0422(6)
H1	0.2219	0.5347	0.6629	0.051*
N2	0.4186(4)	0.4404(2)	0.5234(4)	0.0490(7)
O1	0.1970(4)	0.4948(2)	0.3756(3)	0.0590(8)

Experimental details

The crystal was twinned and the structure was refined using option HKL 5 of the SHELX program [18]. All hydrogen atoms were placed in calculated positions and refined using a riding model. Methyl C-H bonds were fixed at 0.96 Å, with displacement parameters 1.5 times U_{eq}(C), and were allowed to spin about the C—C bond. Aromatic C-H distances were set to 0.93 Å and N-H to 0.86 Å and their U_{iso} set to 1.2 times the U_{eq} for the atoms to which they are bonded.

Discussion

Urea derivatives are important intermediates in medicinal chemistry [3–5]. Many convenient processes have been reported for the production of substituted ureas [6–11]. Aromatic ureas can be substituted efficiently *via* lithiation chemistry [12–16].

The asymmetric unit consists of one molecule of $C_{10}H_{14}N_2O$. The molecule is not planar as indicated by torsion angles C2—C1—N1—C8 = 13.1(5)° and N1—C8—N2—C10 = −12.2(5)°. N—H···O hydrogen bonds form chains of molecules along [001].

Acknowledgements: The project was supported by King Saud University, Deanship of Scientific Research, Research Chair.

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